Material Properties Analysis after Heat Treatment

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Abstract: *This article addresses the influence of heat treatment and corrosion degradation on the mechanical properties of material. The introduction contains information related to the issue of the steel heat treatment theory. The aim of this article is to verify the influence of recrystallization annealing, hardening and refining on the mechanical properties of high-quality carbon steel 12 050. After selecting a suitable material for this experiment, tensile plates were made by the water jet from sheet metal for the tensile tests which were made on the university blasting device. The experiment compares the influence of corrosion degradation, in particular heat treatments. Hardness, corrosion and tensile strength tests during which also the values of acoustic emissions were recorded, are verified here. Pen test (Hsu-Nielson) was used for the activation of signal. The results of the measurements are supplemented by graphs from the course of the tensile test as well as by an acoustic emission record.*

Keywords*: mechanical properties, heat treatment, tensile test, corrosion*

INTRODUCTION

The purpose of the heat treatment of steels is in particular to achieve the mechanical and technological properties of material. This is a procedure in which the temperatures and chemical composition of metal are controlled. Unlike other engineering technologies, such as e.g. machining, forming etc., there is no change of the shapes of parts during the heat treatment (such a change is undesirable) but only required changes are achieved. There may be other positive effects during heat treatment but also negative effects [7]. The most often used as well as cheapest structural steels are wrought carbon steels. Technological properties (machining, forging, welding etc.) and needed mechanical properties (strength, tenacity, hardness etc.) are achieved usually in these steels only by chemical composition of steel, its forming and in a smaller scale also by its heat treatment [11]. Mechanical properties of carbon metals, in particular 12 Structural steels, can be greatly advanced by means of heat treatment. This steel class is distinguished by a higher purity and more advanced way of production [9]. Fe-C structural diagram describes basic parts of its composition. Austenite is a solid solution of carbon in iron, being located in the gama range at a temperature over 723 °C in the diagram. Austenite is tenacious, well cold formable, the maximum solubility of carbon is 2.06 % at a temperature of 1147 °C. [1]. Ferrite is the low-temperature modification of iron crystallising in body-centered cubic system. It has low strength and hardness, cold formable. Ledeburite is the eutectic mixture of austenite and cementite, being formed in alloys with the content of carbon over 2.14 %. Perlite is formed by the eutectic conversion of austenite into a mixture of cementite and ferrite, it is relatively formable and solid [12].

When annealing without recrystallization, the annealing temperature does not exceed the Acl temperature (except for soft annealing in hypereutectoid steels). Phase transformations are insignificant in this case. The proportion of cementite and ferrite in steel does not change, the concentration and division of lattice defects and the size of internal stress change [18]. Due to the recrystallization annealing in steels, the nodular properties are renewed, the elongated grains are removed and strengthened, new ferritic grains are formed and the abilities of plastic deformation after cold forming are renewed. The annealing temperatures are chosen in the interval 550 up to 700 °C, with a lag at this temperature being usually 1 to 5 hours. If a finer structure is requested, a lower annealing temperature is chosen, when using a higher annealing temperature, the grain becomes coarse [10]. The purpose of hardening is to increase the hardness of steel by creating an unbalanced structure. When hardening, a part is heated to the austenitizing temperature, after retaining at this temperature followed by a cooling at a speed faster than the critical speed. The basic structure for hardened steels is the martensite or bainite structure. The required structures can only be achieved in steels with the content of carbon at least 0.3 %. [13]. Hardening can be divided into two main sorts according to the resulting structure – martensite and bainite hardening. Martensite is formed by the rapid cooling of austenite, it is a strongly saturated carbon solution in carbon α (ferrite). [5]. The time of retention at a given temperature does not depend only on the type of applied technology, but also on the kind of the used material and its thickness. The aim of the retention is to ensure in terms of time the course of all expected structural changes, e.g. transformation of the crystalline lattice, diffusion, dissolution, minority phases precipitation etc. Cooling decides on the final utility properties of the material and therefore it is considered to be the most important phase. The principal is to cool the material from the temperature at which it was being retained to the temperature of the environment. The very structure of material is influenced by the rate of the cooling [8]. The optimum hardening environment is such an environment which enables cooling of respective volumes at a rate exceeding only slightly the critical rate. An excessive cooling rate results in the increase of the level of temperature and structural stresses. The cooling environment should have optimally high cooling efficiency in the area of perlitic transformation and to the contrary a relatively low in the area of martensitic transformation. The cooling efficiency depends in particular on the thermal conductivity, the viscosity of the hardening medium and specific heat and heat of evaporation [4].

MATERIALS AND METHODS

ČSN 12 050 structural steel, suitable for annealing and tempering and surface hardening according to the ČSN 41 2050, was chosen for the experiment. It is a cold-rolled metal sheet for the thickness of 1.5 mm. The ČSN 41 2050 standard also limits the chemical composition of the semi-finished product. The tensile plates were made in Kovo Staněk s.r.o. The specimens were cut according to the drawing by the water jet from sheet metal of 1.5 cm thickness. The water jet must have been used for the production, as the use of laser would have caused undesirable thermal influence of the material. Each specimen was also marked in the upper right corner by stamping the appropriate number. All tensile plates were of the same dimensions and thickness so that the tensile test is performed on identical plates. A scheme of the tensile plate is shown in Figure 1.

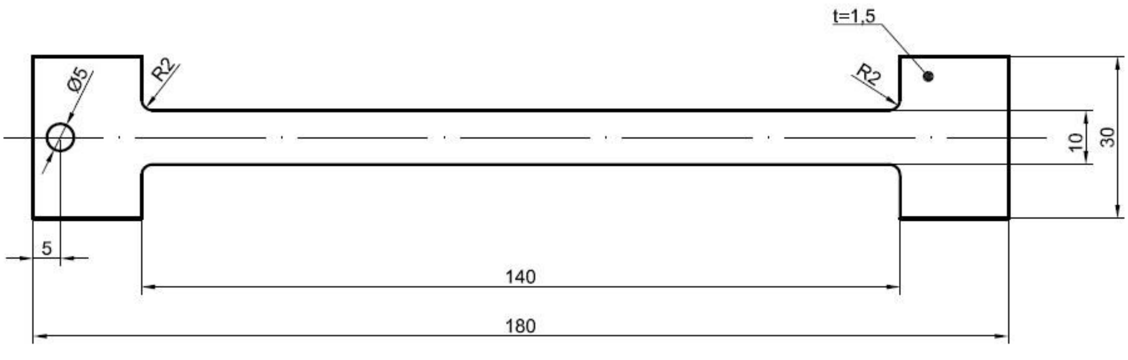

Fig. 1 Scheme of used tensile plate

The tensile plates were divided into four groups of five pieces. Subsequently, each group was thermally influenced by various ways. The hardness of the material was measured on all specimens after heat treatment. A part of the specimen from each group was exposed to accelerated corrosion conditions – salt mist. After 720 hours, test specimens were removed from the corrosion chamber and their weight loss in g×m^2 was determined. This was followed by the tensile test in all specimens, during which the recording of acoustic emission was measured. Tensile strength Rm, contractions Z and elongation A have been determined using the tensile test. The dependencies of mechanical properties of the material on heat treatment and corrosion degradation were compared and evaluated at the end of experiment.

The tested material of 12 050 steel is suitable for hardening and annealing and tempering. For this reason, the below mentioned states of 12 050 steel (1.5 mm metal sheet) were included in the experiment:

1. 12 050.2 Recrystallization annealed (the delivered condition)
2. 12 050.4 Hardened
3. 12 050.6 Annealed and tempered to the lower strength typical for 12 050 steel
4. 12 050.8 Annealed and tempered to the upper strength typical for 12 050 steel

Heat treatment was conducted at the university electrical muffle oven MP05 – 1.1.

Prior to heating, a layer of the Kalsen agent was applied to the specimens. This agent prevents the undesired diffusion and oxidation processes taking place during the hardening, in particular decarbonisation of the upper layers of the specimen. The first group of specimens (10 pieces) was intended only for hardening. The specimens were placed to an oven heated for an austenitizing temperature of 850 °C with the retention of 5 minutes and subsequent rapid cooling in oil. The second group (5 pieces) was hardened in the same way. After a sharp cooling in the oil, the specimens were put again to the oven, now pre-heated for a temperature of 650 °C. The tempering lasted for 1 hour, followed by gradual cooling to ambient temperature. The last group of specimens (5 specimens) was also hardened, but this time tempered at the temperature of 450 °C, again with retention of 1 hour at this temperature and then it was gradually cooled to ambient temperature. A total of 5 HRC hardness measurements were conducted on each specimen. A predetermined number of thermally influenced specimens was placed to a corrosion chamber with a spraying solution of demineralised water and sodium chloride – neutral salt mist (NSS). According to the ČSN EN ISO 9227 standard, the following conditions of the test were kept in the corrosion chamber:

- Temperature: 35 °C ± 2 °C
- Salt solution concentration: 50 g/l ± 5 g/l
- pH value: 6.5 – 7.2

The tensile plates were placed to horizontal stands and put into the corrosion chamber. The stand with the specimens was placed into the chamber so that the specimens are not in the direct direction of the spraying of the spray and at the same time to avoid contact with the inner surface of the chamber or the surface of the adjacent specimen [17]. As recommended by the ČSN EN ISO 9227 standard, the duration of the test was set to be 720 hours during which check was performed regularly. During the tensile test, the record of acoustic emission was made on all specimens. Acoustic emission, hereinafter referred to as AE, belongs to the methods of non-destructive testing of materials. By means of DAKEL – XEDO diagnostic system is possible to analyse the acoustic signals during degradation. This system is an advanced device for capturing and recording of AE parameters, localization of AE sources, and signal sampling. Its main purpose is to monitor periodical tensile test to detect any potential hidden defects in primary circuit technology material and to identify locations that have the highest probability of material defect occurrence. [3]. For acquirement of unified impulses in each case of AE event the Hsu-Nielson pen test was utilized. AE waveguide construction (namely its shape, girth, acoustic conductance) significantly affects the output results in AE signal detection. Waveguides also increase the distance of sensor from signal source, which is also considered unfavourable. It is common in practical application, that installation of sensor directly to surface is impossible, e.g. inaccessible location on construction, or high temperature of measured surface (this is a most common cause of waveguide employment). However, there are certain cases, when employment of waveguide enhances the possibilities of signal detection - typically in structural engineering, namely firm fixing of waveguide into a hole drilled in wood or concrete. Also fixing the waveguide to a plant for transpiration flow measurement in plant stem was conducted [14].

RESULTS AND DISCUSSION

The results of the hardness tests are shown in Table 1.

Table 1 The average measured hardness values

HARDNESS	
OIL HARDENED 850 °C/5 min I. set	Total average [HV]
Specimen No. 6, 7, 8, 9, 10	647,52
OIL HARDENED 850 °C/5 min II. set	
Specimen No. 12, 12, 13, 14, 15	667,88
TEMPERED 450 °C	
Specimen No. 1, 2, 3, 4, 5	356,92
TEMPERED 650 °C	
Specimen No. 16, 17, 18, 19, 20	207,76
RECRYSTALLISATION ANNEALED (the delivered condition)	
Specimen No.21, 22, 23, 24, 25	127,6

Weight loss results

The ČSN EN ISO 9227 standard states that the function of the test chamber is satisfactory if the weight loss of each steel or zinc specimen reaches the prescribed range. The recommended range of weight losses of steel specimens in the NSS test (neutral salt mist) amounts to 70 ± 20 [g · m−2] after 48 hours of the test. For

51 explored specimens, the average weight loss amounted after 48 hours to73, 66 [g· m−2].

Tensile tests' results

For the specimens hardened and tempered at a temperature of 450 °C, material hardness 357 HV and tensile strength of 1131 MPa were measured. The standard states a limit of tensile strength for this hardness amounting to 099 – 1147 MPa. For material hardness 210 HV, which was measured in specimens hardened and tempered at a temperature of 650 °C, the standard limits the tensile strength limit to 647 – 677 MPa, the tensile strength limit of 666 MPa was determined during the tensile test. The both tempering conditions under high temperatures (annealing and tempering) comply with the table values of the ČSN ISO 18265 standard. For recrystallization annealed material (the delivered condition), hardness of 128 HV and the tensile strength limit of 532 MPa were measured. The standard for this hardness in annealed condition states the tensile strength limit of 452 – 470 MPa. The higher measured tensile strength limit than specified in the standard can be explained by the fact that conversion of hardness to the tensile strength limit values is associated with high scattering. One of the reasons of the scattering in uncertainty which can be affected by the changes of microstructure ensuing from heat treatment or cold forming. The standard also specifies that the tensile strength limit table values are only approximate values that cannot replace the tensile test results. The course of the tensile test in specimen tempered at a temperature of 450 °C are shown in Figure 2.

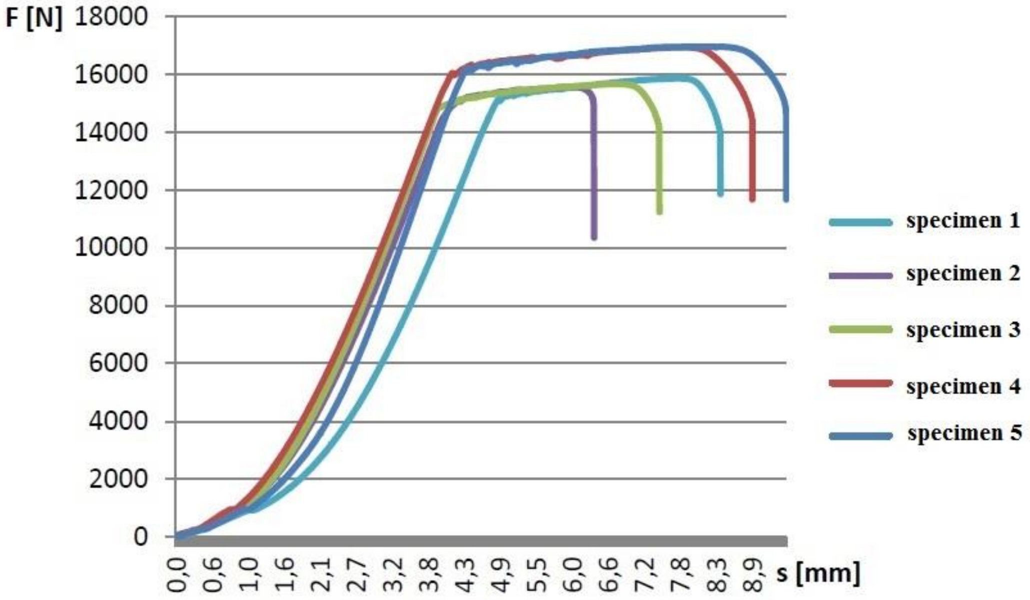

Fig. 2 The course of the tensile test. Specimens tempered at a temperature of 450 °C

The course of the tensile test diagram was in all specimens tempered at a temperature of 450 °C very similar. For tensile plates, there was measured almost the same tensile strength limit as for the hardened specimens was measured, but the tempered specimens achieved a more distinct elongation due to a higher toughness. The yield point is not recorded in the graph. It is more tough material, therefore it was necessary to develop a higher transformation work in order to divide the mass into two parts.

Acoustic emission measurement results

Figures 3, 4 show acoustic emission records during the tensile test in specimens No. 3 and 5.

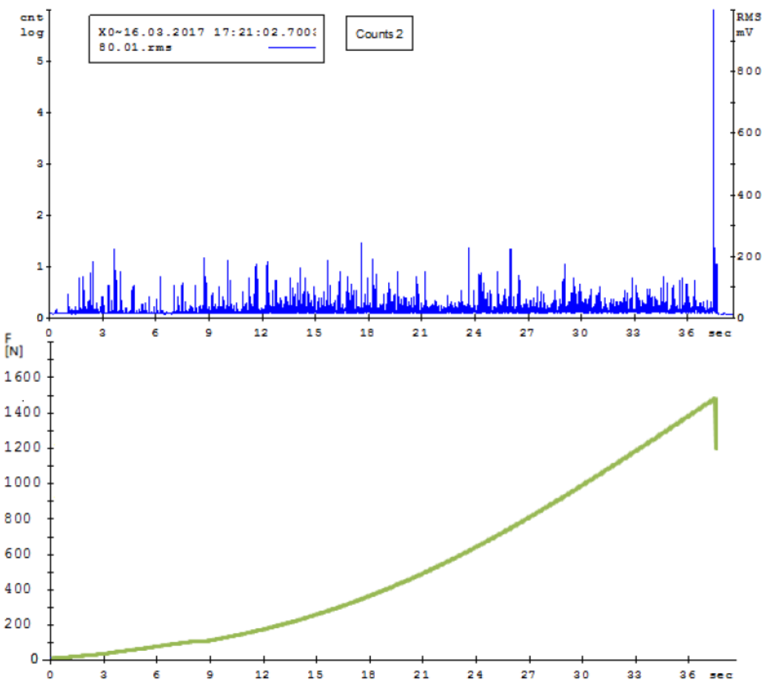

Fig. 3 Acoustic emission record of specimen 3 – oil hardened, austenitization time 5 minutes, exposed to aggressive corrosion environment

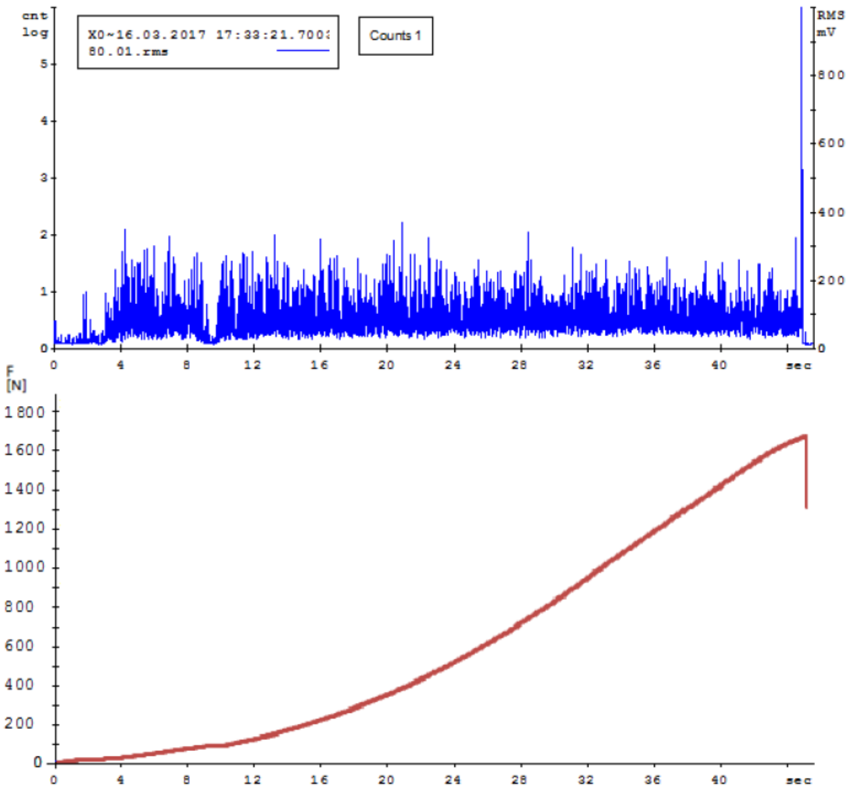

Fig. 4 Acoustic emission record No. – oil hardened, austenitization time 5 minutes

The AE record indicates that RMS designating the overall energy of the AE signal in tempered specimen No.3 exposed to aggressive corrosion environment (NSS) is considerably lower than in hardened specimen No.5 which is not degraded by corrosion. This phenomenon was manifested not only in hardened specimens with the time of austenitization of 5 minutes but also in other heat treatments. The lower overall energy of the AE signal in corrosion degraded specimens can be explained by the fact that the material has been already so degraded and all deformations have been absorbed by microcracks at corrosion pits and points. Intercrystalline and transcrystalline corrosion also disrupted the crystal lattice and caused cracks throughout and among the corns. All these undesirable processes caused a totally lower energy of the AE signal (RMS) in corrosion degradation specimen.

The literature Ptáček (2002) and Kraus (2013) defines that high temperature tempering (annealing and tempering) usually in the range 400 – 650 °C reaches optimum combination of strength, toughness and plasticity while decreasing hardness. The measured data confirm this definition. The specimens hardened and tempered at a temperature 450 °C retained the same tensile strength limit as specimens hardened while considerably decreasing hardness by 200 HV with an increase of toughness and plasticity. For samples tempered at a temperature 650 °C, the tensile strength limit was reduced to 500 MPa with a further decrease of toughness and plasticity.

Černý (1984) states that corrosion negatively affects the mechanical properties of material. This affirmation was confirmed by the performed corrosion test under the effect of neutral salt mist (NSS) lasting 720 hours. It has been also confirmed that steel 12 050 has a low corrosion resistance. The surface of the samples showed 100% degradation already during the first check after 168 hours. Determination of the weight loss did not prove that heat treatment would have a considerable influence on the course of corrosion.

As indicated by the Evans (2011) literature, in today's global competition with short life cycles of products and rapidly changing technologies, customers' demandingness, the companies can only remain competitive by producing quality products. The studies show that quality has a positive influence to financial performance.

CONCLUSION

This article includes a design of measurement methodology, the choice of experimental material, thermal exposure, the hardness test, corrosion degradation of specimens and tensile test supplemented by an acoustic emission record. Practical verification of the mechanical properties of 12 050 structural steel has proved after various heat treatment and corrosion degradation that the properties of this steel can be changed in a wide extent.

The work brings significant information about 12 050 steel in terms of heat treatment and resistance against corrosion. Due to its content and the experiment performed, the work can provide information for further research and application in technical practise.

In particular, an extensive database of knowledge about this material and verification of the influence of corrosion on the mechanical properties has been appreciated.

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